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R. J. Brigham

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INFLUENCE OF MOLYBDENUM ON RESISTANCE TO CORROSION BY PITTING AND CONTACT OF AUSTENITIC STAINLESS STEELS*

R. J. Brigham**

ABSTRACT. The resistance to corrosion by pitting and by contact in solutions of hydrochloric acids of a number of austenitic stainless steels containing 7.5% molybdenum has been evaluated by using the temperature as a criterion. For corrosion by pitting, a critical pitting temperature (CPT) has been defined as being the temperature above which corrosion by pitting takes place and below which the steel is not attacked. The CPT parameter is independent of the rate of increase of the potential and the potential in the case of tests in potentiostatic control and in free corrosion between 10° and 90° C.

The effects of variations in the concentration of chloride and of the small changes in concentration of secondary additive elements (Mn, N, Si, Cu) have been measured. The alloys have been quantitatively classified in relation to their resistance to corrosion by pitting. As an example, stainless steels of the type containing 18% Cr - 0.2% N and containing up to 7.5% Mo and whose nickel content varies between 15 and 25% obey the following equation:

$$\text{TCP (°C)} = 10 + 7 \times \% \text{ Mo}$$

* Presented at the conference "Molybdenum in Special Steels", November 29, 1973, Paris.

** Presented by E. R. Beauchamp.

Resistance to corrosion by contact was measured, and a determination of corrosion by contact (TCC) was made. Tests have shown that the addition of molybdenum considerably reduces the tendency to corrosion by contact, while the effects of the secondary additive elements are considerably less. Correlations between the CPT and TCC data and results from other laboratories and tests in a marine environment are presented.

Introduction

The research program of the Endako Mining Company has focused /128* on the resistance to localized corrosion of austenitic stainless steels containing molybdenum in an oxidizing hydrochloric acid environment. The practical aspects of this program are aimed at the development of alloys having improved local corrosion resistance for application such as pipes for desalination plants and bleaching machinery used for the production of pulp in the paper industry. (The latter requires a large amount of steel in Canada).

The Canadian Research Institute on Pulps and Paper has established, in 1968, that the losses sustained by the pulp and paper /129 plants amounted to 6 - 8% of the maintenance and capital costs of this industry. During the manufacture of the pulp, corrosion attacks mainly the plants bleaching with chlorine and chlorine dioxide. The bleaching solutions contain approximately 1000 ppm of chloride and 300 ppm of chlorine at a temperature up to 70° C. Austenitic stainless steels with molybdenum (particularly type 316L and 317L) are used to reduce corrosion and to improve plant maintenance. Figure 1 shows that these alloys are greatly attacked. This figure shows a 317L type stainless steel belt taken from a washing filter after three months' service in the presence of ClO_2. The apparently undamaged surface hides canal-like fissures that weaken the belt. The operating temperature and the chemistry of the bleaching solutions cannot be modified; therefore, the resistance of stainless steels to

*Numbers in the margin indicate pagination in the original foreign text.

corrosion must be improved. Moreover, the recycling of bleaching solutions, which has as its objective the reduction of effluents, increases the concentration and the temperature of the chlorides. More and more, stainless steels containing 4.5 - 6% molybdenum are used today for service in chlorinated environments. For example, the Uginox NSCD, the Sandvik 2RN65 and the Allegheny Ludlum 6X are used more and more. The Endako Mining company has evaluated the effects of molybdenum and has modified the content of other additive

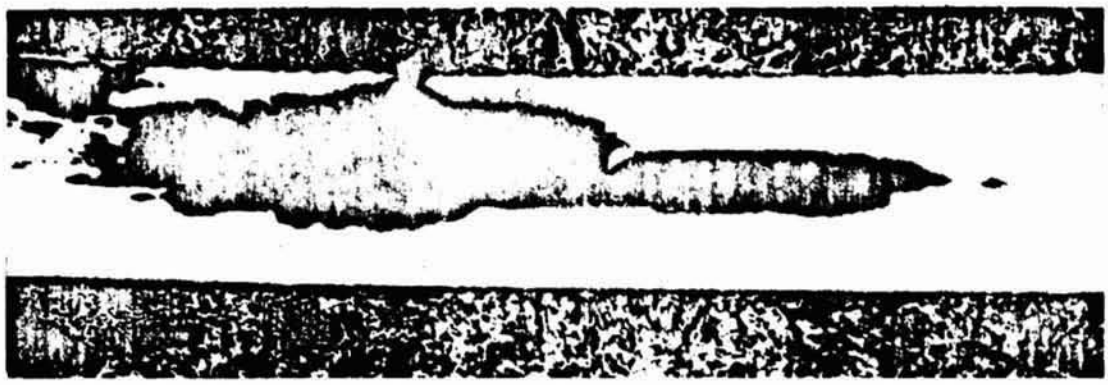


Figure 1. Photomicrograph of 317 stainless steel with pitting and internal corrosion. × 53

elements so as to improve the resistance of austenitic stainless steels to selective corrosion in oxidizing hydrochloric acid.

Alloys Studied

In addition to commercial alloys, a few experimental alloys, whose compositions are given in Figure 2, were prepared in an induction furnace of 20 kg capacity starting from base elements. After deoxidizing with aluminum, the charge was divided into four melts, each containing the desired alloying elements. These were subsequently cast into ingots of 5 kg in sand molds containing CO_2. The ingots were cut into sections and the surface defects were ground off. Afterwards, the ingots were laminated into sheets of approximately 2 mm thick*. The specimens were heat treated and ground with

*The rough structure of the ingots caused some difficulties.

silicon carbide paper (120) after one hour of annealing at 1093° C. The precise composition of the alloys is given in Table I.

Alloy No.	1	2	3	4	5	6
Melt No.	7579	7580	7581	7582	7584	7585
% Cr	18	18	18	18	18	18
% Ni	20	20	20	20	10	10
% Mn	1	1	1	1	10	10
% N	0,04	0,2	0,04	0,3	0,3	0,3
% Si	0,3	0,3	0,3	1,5	0,3	0,3
% Cu	-	-	2	-	-	2
C < 0,03		S < 0,015			P < 0,015	
Molybdenum content 3 - 4,5 - 6 - 7,5 %						

Figure 2. Composition of alloys studied

Potentiostatic polarization for measurement of resistance to corrosion by pitting

The anodic polarization of an alloy in an appropriate electrolyte using a potentiostat is more and more accepted as a method for measuring the resistance to corrosion by pitting. Figure 3 shows a typical curve for a stainless steel during anodic polarization and shows its S shape. In an electrolyte such as sulfuric acid, the entire curve is obtained up to very noble potentials. In contrast, in the presence of Cl^- ion, an increase in the current density at a lower potential (pitting potential) is sometimes observed because the onset of pitting corresponds to an increase in the current density. The potential that can be assigned to the oxido-reduction system H^+/H_2, Fe^{+++} and Cl_2/Cl^- is shown in Figure 3.

Stainless steels containing molybdenum are subjected to anodic polarization effects in stages (a jump of 50 mV every 2.5 minutes) in a 3.5% acidic solution (pH 3) of NaCl at a temperature of 27° C. The results for the alloy groups containing 18% Cr, 20% Ni, and 0.2% N are shown in Figure 4. At this temperature, the alloys containing 4 - 7.5% molybdenum seem identical. On the other hand, the alloy containing 3% molybdenum is less resistant. The temperature effect was measured so as to distinguish between alloys of the same group. /130

TABLE I. CHEMICAL ANALYSIS IN %**

Alloy	Cr	Ni	Mn	C	N	Si	P	S	Mo
7466-2	19,44	20,23	0,09	0,03	0,22	0,33	0,016	0,010	4,45
7466-4	18,00	18,93	0,08	0,07	0,19	0,32	0,019	0,015	7,40
7579-1	19,32	20,46	0,99	0,02	0,06	0,50	0,010	0,015	3,07
7579-2	—	—	—	—	—	—	—	—	4,56
7579-3	—	—	—	—	—	—	—	—	6,18
7579-4	17,64	18,32	0,90	0,02	0,06	0,48	0,011	0,015	7,56
7580-1	19,17	20,24	0,99	0,03	0,19	0,44	0,012	0,012	3,04
7580-2	—	—	—	—	—	—	—	—	4,48
7580-3	—	—	—	—	—	—	—	—	5,60
7580-4	17,88	18,96	0,89	0,03	0,20	0,43	0,012	0,013	7,30
7903-1	18,89	15,13	*	0,02	*	*	*	*	4,05
7995	18,67	23,8	0,46	0,043	0,224	0,33	0,013	0,008	6,3
8426	19,38	22,68	0,38	0,04	0,20	0,47	0,014	0,014	6,04
8429-1	17,95	12,71	0,53	0,03	0,07	0,71	0,012	0,012	2,50
8429-2	—	—	—	—	—	—	—	—	2,75
8430-2	17,74	12,63	0,46	0,03	0,23	0,58	0,014	0,013	2,85
8430-3	—	—	—	—	—	—	—	—	3,26
304	18,08	8,60	*	0,06	*	*	*	*	0,05
316L	16-18	12,33	*	< 0,03	*	*	*	*	2,47
317L	18-20	14,03	*	< 0,03	*	*	*	*	3,11
Alloy A	17,3	23,8	1,72	0,018	0,034	0,44	0,010	0,004	4,60
Alloy B	15-17	24-27	< 2,0	< 0,08	0,10-0,20	< 1,0	*	*	6,0

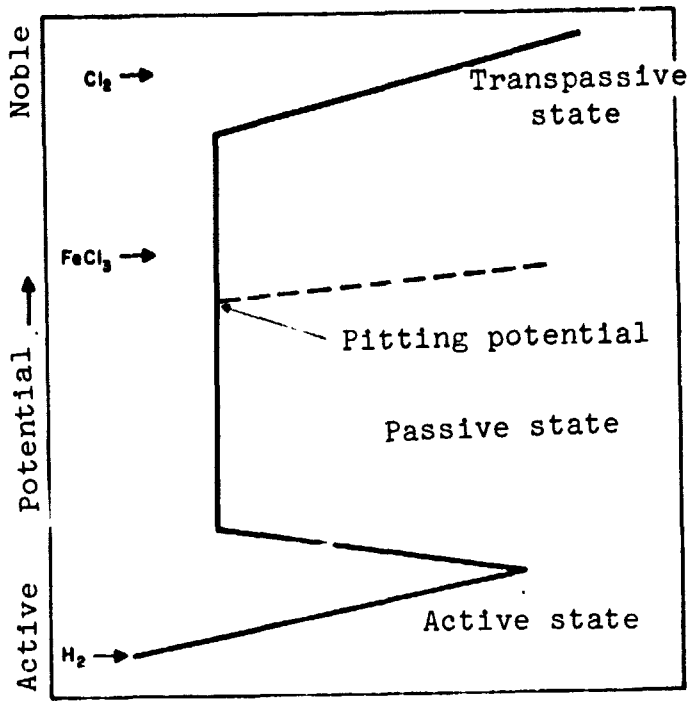
— not analyzed but divided melt.

* not analyzed.

** Commas represent decimal points.

The temperature was chosen as a criterion because most industrial processes are operated above ambient temperatures.

Figure 5 shows the results of the anodic polarization of the 7580-3 alloy (5.6% Mo) for different temperatures. In the range from 10 to 35° C, the alloy seems to attain the transpassive state at noble potentials and the temperature effect is not very pronounced. Likewise, in the range from 60 to 90° C, corrosion by pitting appears at very active potentials. The results at 50° C suggest a transition state between the low and high temperatures. The potential needed to reach a current density of 10 $\mu A/cm^2$ was chosen as a criterion for corrosion by pitting so as to distinguish between the behavior of those alloys in the transpassive state and those that are attacked. In Figure 5, this potential approaches the pitting potential as defined



Current density or corrosion rate (logarithmic scale)

Figure 3. S-shaped anodic polarization curve for a stainless steel with and without pitting

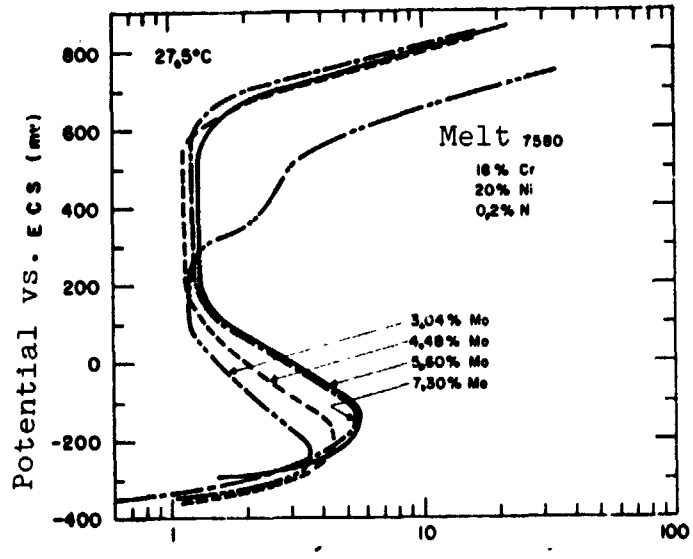


Figure 4. Experimental curves for anodic polarization for stainless steel containing molybdenum exposed to a 3.5% NaCl solution

in Figure 3. The measurement data of pitting potentials obtained from anodic polarization curves for the 7580 alloy series are presented in Figure 6 by a series of Z-shaped curves. Each experimental point represents the result of anodic polarization curve of the type shown in Figure 5. This figure clarifies the doubtful results previously obtained with measurements at ambient temperature. It is obvious that the behavior of alloys at low and high temperatures cannot be separated. At low temperature, all alloys are in the transpassive state, whereas at high temperatures their pitting is produced at very active potentials. Nevertheless, at intermediate potentials varying from 300 to 700 mV_{ECS}, the resistance of alloys to corrosion by pitting can be evaluated by using the temperature as a criterion.

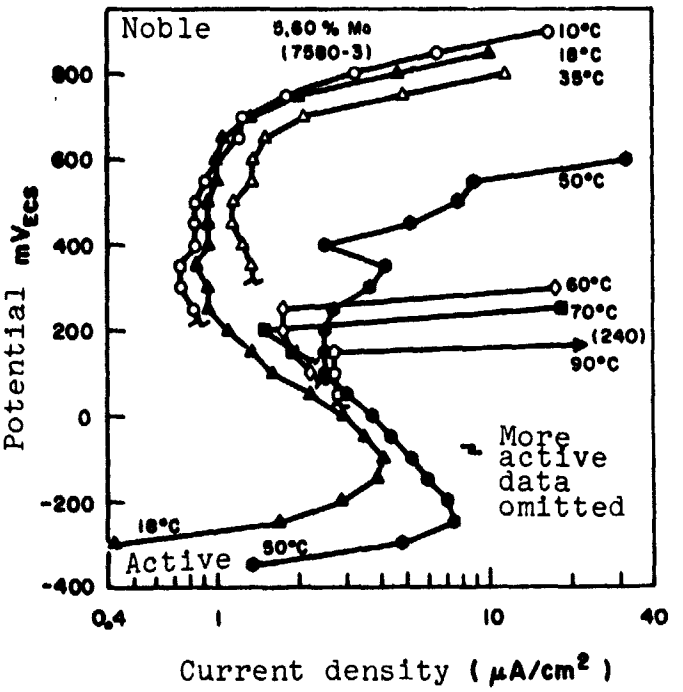


Figure 5. Anodic polarization of alloy 7580-3 (5.6% Mo) at different temperatures

Temperature as a criterion for corrosion by pitting

Unfortunately, these data were obtained at high rates of increase of the potential (1200 mV/h), and their representation of real service conditions is not immediately obvious. Nevertheless, the published data show predictable tendencies with regard to the polarization curves obtained at lower rates of increase of the potential. As far as the alloys having pitting potentials in the general region of the plateau of the lower potential (Figure 6), Leckie and Uhlig [4] have found that the potential for corrosion by pitting /131

became more active when the rate of increase of the potential decreased. Bond and Lizlovs [5] have found that the alloys whose pitting potential is in the general region of the plateaus of the upper potential in Figure 6 had a nobler potential for corrosion by pitting when the rate of increase of the potential decreased. This is shown by arrows in Figure 7. The solid line represents the results of the potential for corrosion by pitting for the case of a potential increase rate of 1200 mV/h as a function of temperature. The broken line represents the curve suggested for an infinitely high rate of increase of the potential (long exposure times). This is based on the trends discussed earlier. The proposed curve suggests that the temperature is a reliable criterion to indicate the resistance to corrosion by pitting, whatever the potential, so long as it lies between the upper and lower plateaus of the potential.

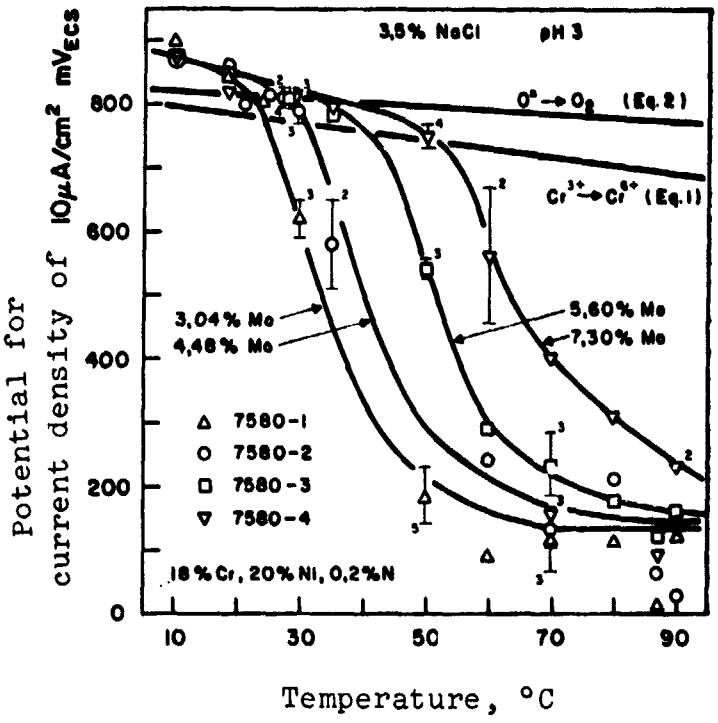


Figure 6. Effect of molybdenum content and temperature on the potential needed to produce a current density 10 μ A/Cm² for steels containing 18% Cr, 20% Ni, and 0.21% N

Experimental proof

Three experimental proofs are discussed to support the existence of the curve proposed above.

Two-phase alloys

A set of single- and two-phase stainless steels containing molybdenum, depending on heat treatment, was subjected to a potentiostatic test such as described above. In Figure 8, the results for single-phase alloys (solid line) and two-phase alloys (broken line) are shown in the same way as in Figure 6. The nefarious effect that the second phase has on the resistance to corrosion by pitting will be discussed below. Nevertheless, let us look at the effect of the second phase on the shape of the curves. The second phase produces favorable heterogeneous nucleation centers and, under these conditions, the curves of the potential for corrosion by pitting as a function of temperature nears the vertical direction.

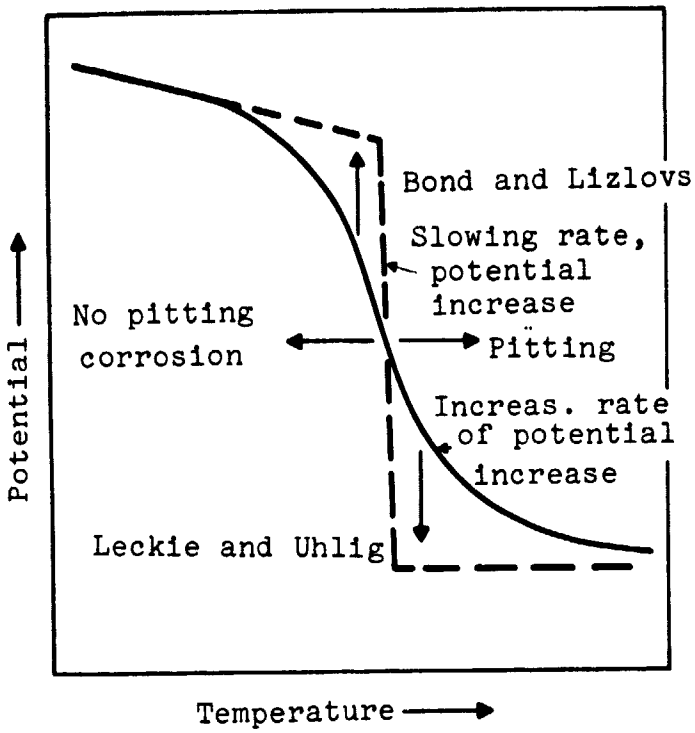


Figure 7. Hypothetical curve showing the effect of the rate of increase of the potential and temperature on the potential needed to initiate pitting corrosion of stainless steels

Tests with variable temperature

With a fixed potential of 500 mV_{ECS}, the temperature of the solution in contact with the sample is increased until pitting is observed. In all cases, an excellent correlation exists between the results labeled T_{500}; the results of tests with increasing potential are shown in Figures 6 and 8.

Tests of total immersion in solution with different oxidizing power

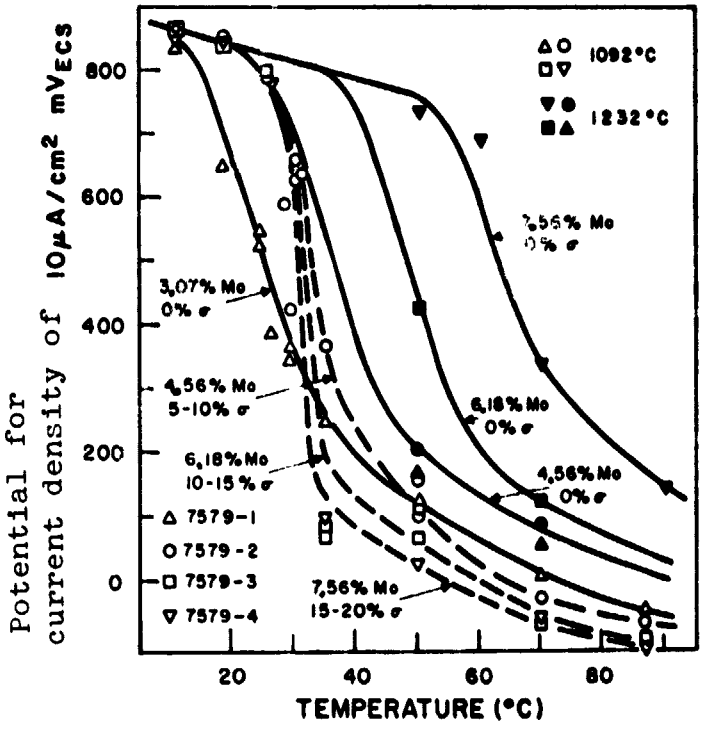


Figure 8. Effect of molybdenum content on potential needed to produce pitting corrosion using an increase in the current density 10 μ A/Cm² as a criterion for alloys of 18% Cr and 20% Ni exposed to a solution of 3.5% NaCl and a pH of 3

single phase alloys — solid line; two-phase alloys (broken line)

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If, for each alloy, the vertical potential curves as a function of the proposed temperature are correct, any critical temperature must exist. Above this temperature, corrosion by pitting would take place, and below it the alloy would not be attacked if it were immersed in a hydrochloric acid with an intermediate oxidizing power. In Figure 9, corrosion of 317L stainless steel is measured as a function of temperature, but immersed in three similar solutions containing chlorine ion but of variable oxidizing power. The samples were immersed in the solutions for 24 hours at a constant temperature. In the absence of corrosion by pitting, the temperature was increased by 2.5° C, and the samples immersed anew. The criterion of corrosion by pitting used was the formation of one pit on a plane surface. Other forms of corrosion by pitting, such as those found on edges, were not considered. Figure 9 shows that 317L type stainless steel is not attacked by the solutions at 27.5° C, but the corrosion by pitting in all solutions is observed when the temperature is raised to 30° C, such as shown by the "u" symbol on the figure (the size of the "u" indicates the error margin). Tests extended to 60 hours gave identical results.

Critical Pitting Temperature

The critical pitting temperature (CPT) is defined as the transition temperature above which corrosion by pitting takes place, and below which no corrosion occurs. Figure 9 shows that the CPT is independent of the potential (in the intermediate interval), and is easily determined by immersion in a solution of 10% $FeCl_3$ for 24 hours. This method also shows that the results are also independent of chlorine ion content between 4 and 10% $FeCl_3$, as illustrated by Figu .

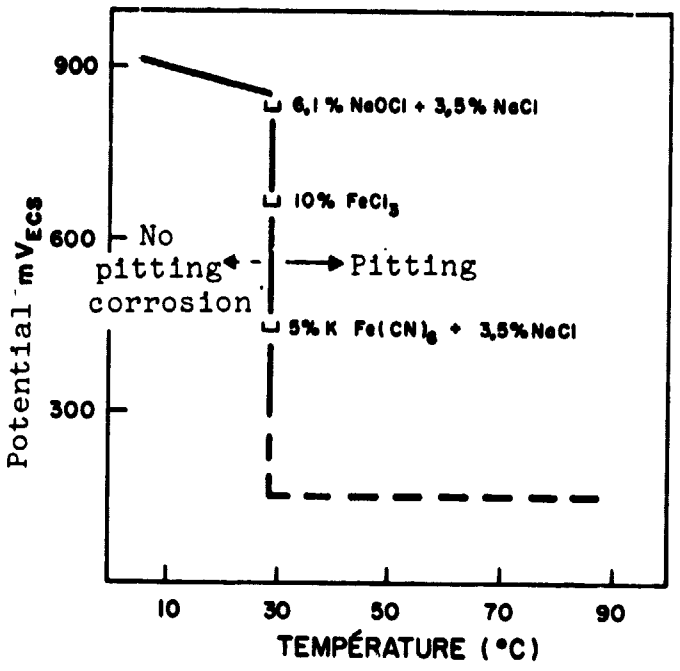

Figure 9. Corrosion by pitting of 317L stainless steel exposed for 24 to 66 hours to solutions of various corrosive power

When the $FeCl_3$ content decreases, the CPT increases for tests from 24 to 66 hours. The increase in immersion time for lower concentrations of $FeCl_3$ is necessary so as to exceed the incubation period preceding the onset of corrosion by pitting. The above tests show that the CPT is independent of the chlorine ion potential and of the immersion time. Then the CPT was used to determine the effects of the alloying elements on the resistance to corrosion by pitting of austenitic stainless steels.

Effects of Molybdenum

In Figure 11, the CPT is shown as a function of the content of molybdenum for commercial and experimental austenitic stainless steels containing 18% Cr and 0.2% N, and having a nickel content

varying between 12 - 25%. The behavior of austenitic steels is expressed by the following equation:

$$TCP\ (^{\circ}C) = 10 + 7 \times \%\ Mo$$

To the left of the curve in Figure 11, corrosion by pitting does not take place, whereas at temperatures located to the right of the curve, corrosion is probable. For example, a sample of 7580-2 (4.48% Mo) steel was immersed on May 26, 1972, in a 10% $FeCl_3$ solution at temperatures as high as 35° C. The curve on Figure 11 shows that there should be no corrosion and that the surface should remain intact because the CPT for this alloy is in the range from 42.5 to 45° C. Molybdenum is the primary element for increasing the resistance to corrosion by pitting and, consequently, this element was used to compare the CPT of alloys of varying composition.

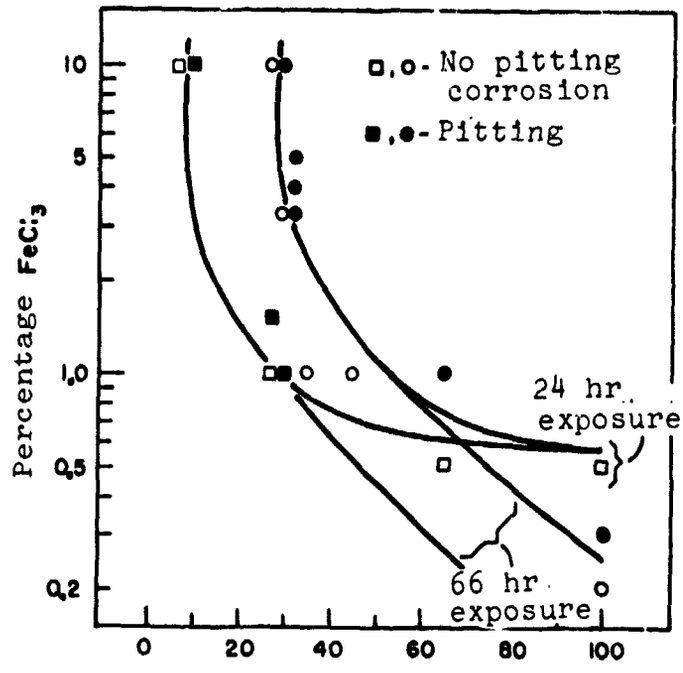

Figure 10. Effect of $FeCl_3$ concentration and of exposure time on the temperatures of corrosion by pitting of 304 and 317L stainless steel

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1) Effect of nitrogen

Figure 12 shows the results obtained for a similar group of alloys, but with a lower nitrogen content. For single-phase alloys, the behavior is described by the following expression:

$$TCP\ (^{\circ}C) = 5 + 7 \times \%\ Mo$$

A lower nitrogen content (0.06% compared to 0.2% N) seems to slightly reduce the resistance to corrosion by pitting. Nevertheless, the principal effect of nitrogen is to stabilize the austenite. The alloy becomes two-phase after annealing at 1093° C when nitrogen is absent and the molybdenum content is increased. However, after a heat treatment at a temperature of 1232° C, the austenitic phase is completely recovered. As shown in Figure 12, the presence of the second phase limits the resistance to corrosion by pitting of two-phase alloys by making the second phase rich in molybdenum. In this case, the resistance to corrosion by pitting is not reduced, but only limited.

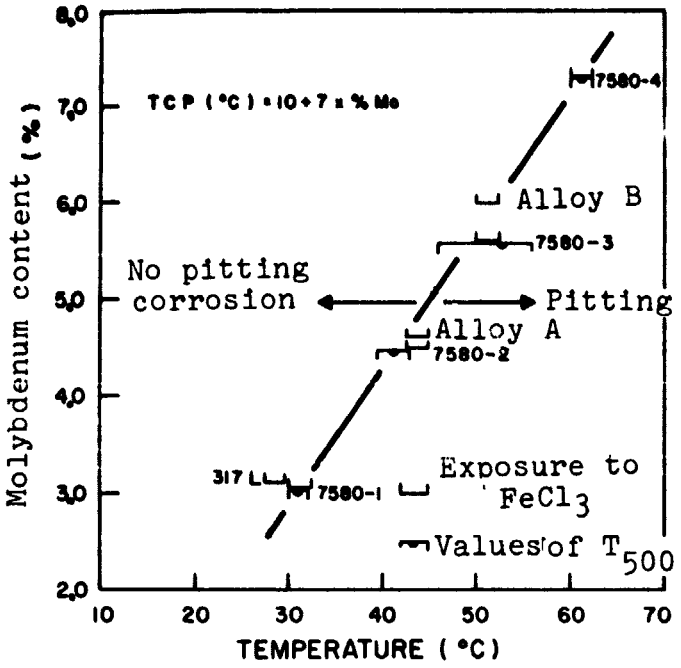

Figure 11. Critical pitting temperature (CPT) as a function of molybdenum content for experimental and commercial austenitic alloys

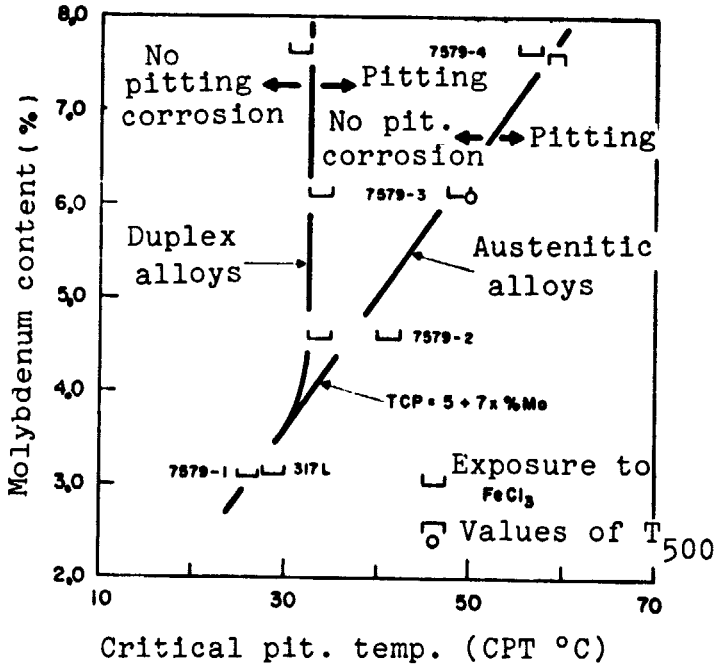

Figure 12. CPT as a function of molybdenum content for single- and two-phase stainless steel alloys

II) Effect of silicon

The preliminary results with an alloy group having a low nitrogen

content but containing 1.5% Si can be expressed by the following expression:

$$TCP\ (^{o}C) = 15 + 7 \times \%\ Mo$$

This is in agreement with the results of Tomashov et al. [6], who have found that the resistance to corrosion by pitting for steels containing 18% Cr and 14% Ni, exposed to a 9% $FeCl_3$ solution at ambient temperature, was inproved by increasing its silicon content.

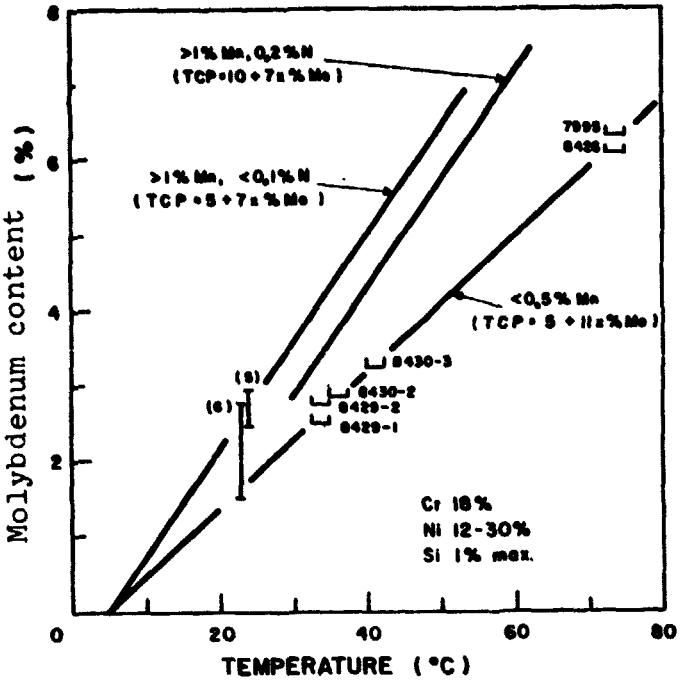

Figure 13. CPT as a function of molybdenum content for groups of stainless steels of various chemical compositions

III) Effect of copper

Only negligible effects on the CPT were observed when the copper content was varied between 1.5 and 2%.

IV) Effect of manganese

All alloys shown on Figures 11 and 12 have a manganese content between 1 and 2% (the AISI specifications require a maximum of 2% Mn). Resistance to corrosion by pitting is somewhat improved when lowering the manganese content to 0.5%. Figure 13 shows the resistance to corrosion by pitting for alloys with low manganese content. These alloys are represented by the equation:

$$TCP\ (^{o}C) = 5 + 11 \times \%\ Mo$$

A decrease in manganese content and an increase in nitrogen to compensate for the loss in austenization brings about a synergistic and significant improvement in the resistance to the onset of pitting /134 in austenitic stainless steels containing molybdenum. A similar behavior was noted for alloys containing 10% Mn.

Additionally, in Figure 13, all the CPT data are schematically presented with the published results on the resistance to corrosion by pitting of austenitic stainless steels exposed to $FeCl_3$ solution. Bond and Lizlovs [5], as well as Tomashov et al. [6], have observed that a group of alloys containing different amounts of molybdenum exposed at a constant temperature in a solution of $FeCl_3$ require a minimum amount of molybdenum to stop corrosion by pitting. These results are shown in Figure 13, and agree well with the data obtained.

Corrosion by contact

The above data on CPT are related to the conditions under which a pit is initiated on austenitic stainless steel. The CPT is neither a function of the chloride content between 4 and 10% $FeCl_3$, nor of the potential and the immersion time. The CPT can thus play a very important role in the conceptualization of apparatus exposed to dilute solutions of chlorine ions that could become concentrated because of evaporation. However, in most practical applications, corrosion by contact (a phenomenon similar to the growth of pits) limits the use of stainless steels. Unfortunately, today, no criterion is universally accepted by the classification of the resistance of alloys to corrosion by contact. Moreover, the effects of a number of variables, such as the potential and the composition of the solution, are not well understood. Thanks to the success obtained with the classification of alloys with the CPT, a logical extension of this approach indicates a method for evaluating the corrosion by contact as a function of temperature and exposure to the same solution as those used during the tests for corrosion by pitting. Three types of fissures were used to ensure that the alloys were subjected to extreme conditions: metal on metal, teflon on metal, and rubber band on metal.

The samples were immersed for 24 hours at each temperature, and examined for indications of corrosion by contact. The temperature was increased by intervals of 2.5° C until corrosion by contact appeared in one of the forms of corrosion described above. The temperature interval so determined thus defined the limit to the resistance to corrosion by contact.

The preliminary results, including a curve of the corrosion by pitting, are shown in Figure 14. In all cases, the contact corrosion temperature (CCT) is less than the CPT, regardless of the molybdenum content. The only alloying element that improved the resistance to corrosion by contact was molybdenum, contrary to the above discussion concerning corrosion by pitting. All austenitic alloys conform to the following expression:

$$TCC\ (^{\circ}C) = -50 + 12.5 \times \%\ Mo$$

Apparently a pit once initiated at the CPT or above cannot be stopped so long as the temperature is not lowered below the critical corrosion by contact temperature (CCT).

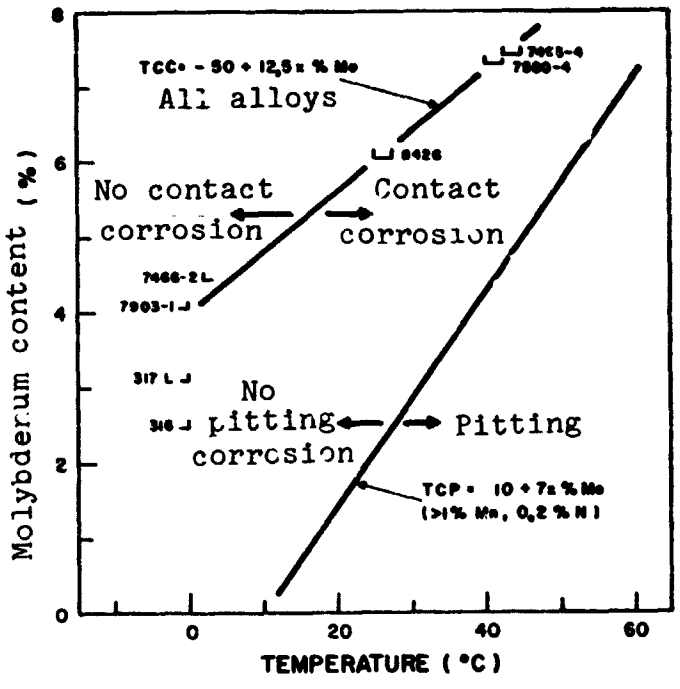

Figure 14. CPT curve for a group of stainless steel, and CCT curve for various stainless steels as a function of molybdenum content

Although the parameters chosen for this study are extreme for these tests, a classification of alloys based on practical applications was obtained.

Conclusion

A critical pitting temperature has been defined for austenitic /135
steels containing molybdenum and 18% Cr. This gives a quantitative measure of their resistance to pitting. In all cases, the molybdenum content considerably increases the resistance to localized corrosion. The other additive elements have a beneficial or synergistic effect when present with molybdenum. Molybdenum will become an indispensible element for increasing the resistance to corrosion of stainless steels that will be exposed to hydrochloric acid solutions at high temperatures.

Continued research in this area is thus stimulated by the excellent correlation that exists between the results obtained and the immersion test results obtained by Kügler, et al. [7]. During the three year immersion tests in sea water near Heligoland, these investigators have found that an increase of 2.8 to 3.3% molybdenum in austenitic alloys of the type containing 18% Cr has effectively eliminated the localized corrosion.

Acknowledgement

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Discussion

Mr. Montuelle. Thank you very much, Mr. Beauchamp. Is there anyone who wishes to ask questions about the subject presented by Mr. Beauchamp?

Mr. Defranoux. — I would like to ask a question concerning the effect of manganese: You have pointed out that the decrease in the content of manganese is suitable for increasing the critical temperature. May I ask you if you have correlated this effect to a structural effect, or rather to an effect of sulfur composition such as was shown in an American study, I believe, last year? Sulfurs of manganese correspond to high manganese content, whereas for low manganese content, the sulfurs are rich in chromium and are less toxic. May I ask your opinion on this subject?

Mr. Beauchamp. — I believe it is mostly the sulfur effects.

Mr. Farge. — Mr. Brigham would like to add that, in the case of steel containing from 0.5 to 20% manganese, the sulfurs are of the spinel type (Fe, Mn) Cr_2S_4, whereas in the case of steels containing less than 0.5% manganese, the chromium sulfurs (CrS) are formed. Apparently, the sulfurs of the spinel type cause the corrosion by pitting more easily than the sulfurs of the chromium type.

Mr. Hanon. — I would like to know if the surface condition has /136
any effect on the critical pitting temperature. In other words, is an electrolytic polishing better than a mechanical one?

Mr. Beauchamp. — We have used as a criterion for corrosion by pitting the formation of a pit on a plane surface. The other forms of corrosion by pitting, such as found on edges, were not considered.

Mr. Hanon. — What is the preparation and the state of the surface of your samples?

Mr. Farge. — According to Mr. Brigham, the samples were worn by friction on silicon carbide of grain size 120. The effect of electrolytic polishing compared to mechanical polishing was not studied.

Mr. Evrard. — Do you also have corrosion tests on soldered joints?

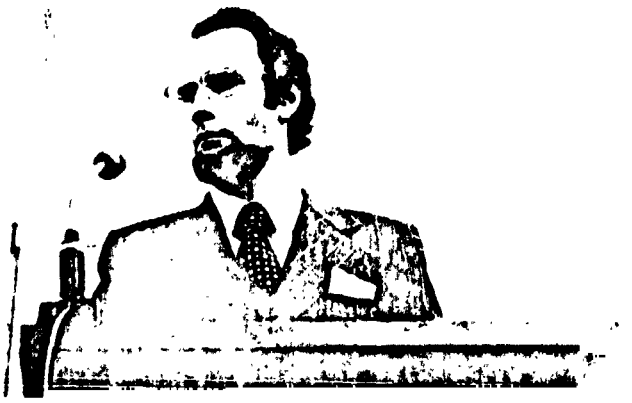
Mr. Beauchamp. — No, but it is provided for in our research program.

Remark. — To complement this question: All the tests that you have carried out to obtain these curves were on samples taken from a sheet. It is evident that when you have a soldered joint, the curves would be slightly different. Do you think that the soldered joint would be better than the sheet itself to obtain approximately the same results?

Mr. Farge. — According to Mr. Brigham, in this case, this will be a lot worse because of the segregating alloying elements and the formation of a second phase such as the sigma phase or the ferrite.

Mr. Montuelle. — I would like to ask you about the synergistics of molybdenum-copper. There is only a brief mention in the text concerning the part played by coppe associated to that of molybdenum.

Mr. Beauchamp. — Mr. Brigham says that he has not found any effect.



Mr. Beauchamp

Mr. Montuelle. — Has there been no effect on the samples free of copper?

Mr. Farge. — Apparently it is the same with or without copper. Mr. Brigham has also tried commercial steels that contained copper, and he realized that they behaved in the same manner.

Mr. Montuelle. — Are there any other questions for Mr. Beauchamp and Mr. Brigham?

Mr. Charbonnier. — How can the beneficial action of molybdenum on the resistance to corrosion by pitting and contact be explained?

Mr. Farge. — For the moment, Mr. Brigham does not propose a mechanism. He simply presents observations concluded from his experiments.

Mr. Montuelle. — I very much thank Mr. Brigham and Mr. Beauchamp.

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